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Effect of temperature and additives on the yields of products and microwave pyrolysis behaviors of wheat straw

Xiqiang Zhao, Min Wang, Hongzhen Liu, Chao Zhao, Chunyuan Ma, Zhanlong Song*

National Engineering Laboratory for Coal-fired Pollutants Emission Reduction, Shandong Provincial Key Lab of Energy Carbon Reduction & Resource Utilization, Shandong University, Jinan 250061, China

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ABSTRACT

The microwave pyrolysis of wheat straw has been studied in the laboratory using a fixed-bed microwave reactor. The effects of both the temperature and the presence of additives on the yields and properties of products and pyrolysis behaviors are the focus of this work. The results showed that at constant temperature (500 °C) pyrolysis, the yield of solid products decreased with the increase in microwave power during the heating up stage, whereas the yield of gas products was the opposite; H_2 concentration increased by approximately 20 vol.% with the increase in microwave power, leading to a high heating rate. The lowest concentration of CO_2 was obtained when adding CaO. The yields of H_2 and CO increased with the promotion of gasification reactions between carbon and CO_2 and the dry reforming and cracking reaction of CH_4 under high temperature by K_2CO_3. Two gas release peaks occurred when the sample continued to be heated after pyrolysis at a constant temperature. The first peak was the release of most volatiles, and the second one was the release of residual volatiles after temperature control canceled. The final temperature and presence of additives had strong effects on the yields and properties of the final products.

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1. Introduction

The utilization of biomass is gaining more and more interests due to the increasing environmental concerns and energy security challenges. Among various utilization patterns, pyrolysis is one of the most promising thermo-chemical conversion routes. In the pyrolysis process, primary reactions are concerned with the degradation of biomass into char and numerous volatiles, whereas secondary reactions involve primary volatiles as they move through the high-temperature char layer and/or the reacting environment. Generally, secondary reactions occur inevitably, and more gaseous and solid products are obtained [1]. For obtaining more valuable products and convenient for operation, the size of sample should not exceed 1 mm in order to obtain a high heating rate [2]. As a result, the pretreatment of raw materials especially crop straws can be energy-intensive. Based on considerations above, present researches on pyrolysis are focusing on improving pyrolysis conditions and achieving directional transformation to the targeted-products by adding catalysts and/or adopting new heating methods such as plasma heating, microwave heating and so forth [3,4].

Microwave heating has merits of "internal heating", so the large-sized sample can be heated to reduce the pretreatment processes of raw material. Consequently, microwave pyrolysis attracts increasing interests of more and more researchers. Recently, it has been employed to pyrolyze wood block, coffee hulls and straw bale, and valuable results different from conventional heating has been obtained [5–7]. In addition, the catalytic effects of additives on microwave pyrolysis of pine sawdust [8] have been studied. It found that all the eight additives have increased yield of solid products greatly, and decreased yield of gaseous products more or less, but all additives have made these gases evolve earlier. The microwave-induced pyrolysis of rice straw has also been studied [9,10], and the results indicate that the major compositions in gaseous product were H_2, CO_2, CO, CH_4 of 55, 17, 13, 10 vol.%, respectively. Other researches on microwave pyrolysis of biomass include pyrolysis of pine pellets [11], waste tea [12], corn stover [13,14], wheat straw [15] and oil palm biomass [16]. In general, the gases contained a high H_2 content, implying that microwave pyrolysis produces not only high-rich fuel gas but higher than under conventional heating.

For in-depth exploration of mechanisms of microwave pyrolysis, it is important to probe the impacts of temperature on microwave pyrolysis and some researches have been carried out, such as microwave pyrolysis at constant temperatures completed by Wang et al. [17]. However, the influence of temperature on pyrolysis is complex and more researches are necessary. In

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* Corresponding author. Tel.: +86 531 88399372; fax: +86 531 88395877.
E-mail address: zlsong@sdu.edu.cn (Z. Song).

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Table 1
Ultimate and proximate analyses of samples (wt.%, ad).

M_{ad}	A_{ad}	V_{ad}	FC_{ad}	C_{ad}	H_{ad}	N_{ad}	S_{ad}	O[a]
7.4	5.9	69.3	17.4	41.41	5.86	1.44	0.17	45.22

M: water content, V: volatiles, A: ash, FC: fixed carbon; ad: air-dried basis; [a]subtract.

addition, for obtaining more targeted products and reducing the tars, additives are often added to the samples [18–20]. Furthermore, the high CO_2 content in gas products limits the utilization of the gaseous products, and CO_2 reforming of CH_4 is adopted to reduce the content of CO_2 [21]. But few investigations on the microwave pyrolysis characteristics of samples mixed with additives, like catalysts for tar cracking and absorbent for CO_2, has been carried out.

In this paper, a special microwave pyrolysis reactor (MWR) was used, which was designed for investigation on microwave pyrolysis at a constant temperature or a constant microwave power, helping explore the mechanism of microwave pyrolysis. Our work aimed to study the microwave pyrolysis of wheat straw at constant temperatures, focusing on the influences of temperature, microwave power, and additives on the product yields, compositions, and fuel properties.

2. Materials and methods

2.1. Materials

The biomass used in this study was wheat straw, collected from Liangshan County, Shandong Province in Eastern China. The wheat straw was naturally air dried under sunlight for 30 day to get rid of the surface water. Then it was shredded, grinded and sieved to 0–200 mesh (0–0.090 mm) in preparation for the sample. The density of wheat straw powder was about 300 kg/m^3. The results of the proximate and ultimate analysis of the wheat straw sample are listed in Table 1. The volatile content of wheat straw is 69.30 wt.%, supporting the use of wheat straw as an energy source.

The chemical structures of wheat straw are identified by Fourier transform infrared spectrometer (Tensor 27, Bruker, Germany). Wheat straw contains a large number of oxygen-containing functional groups such as OH (3400–3200 cm^{-1}), C—H (2936–2916 cm^{-1} olefin), C=O (1765–1715 cm^{-1}), C—O—C (1270 cm^{-1}), CO—(H) (1050 cm^{-1})) alkyl, ether, aromatic, ketone, and alcohol (3000–2800 cm^{-1} and 900–700 cm^{-1}), indicating the high-oxygen content of the sample.

2.2. Experimental apparatus

A schematic diagram of the MWR is shown in Fig. 1. The MWR mainly consists of five components: a modified 3000 W, 2.45 GHz domestic microwave oven, a 2-figure balance (Shanghai, Yousheng BS1000G), a thermocouple for temperature measurement, a circulation heating device, a product cooling and collection system, and a computer running proprietary acquisition and control software. To prevent the magnetron from being damaged by the reflected microwave, a circulator is built between the microwave source and the MWR. Thus, the reflected microwave is absorbed by the circulating water.

The reaction tube (18 cm length, 8 cm outer diameter) and crucible are both made of quartz. Both ends of the quartz tube were sealed tightly with a silica o-ring and a PTFE sealer. The accurate measurement of the temperature of the sample is crucial in thermal studies. Metal objects, including wires, cannot normally be placed in a microwave field without either severely distorting it or causing electrical discharges. However, in confirmation of the findings of [22,23], it is possible to use thermocouples in a microwave oven provided they are thin, have a grounded metal sheath, and are held exactly at 90° to the electric field component of the microwave energy. In the present study, 1-mm diameter, stainless steel sheathed type K (chrome-alumel) thermocouples are used, covering the temperature range of 0–1000 °C.

The temperature control function was introduced to carry out microwave pyrolysis of sample at constant temperatures. The system allows setting of a constant microwave power or the power can be adjusted based on a desired temperature value. Thus, when the temperature of the sample reached the target temperature, the microwave power was reduced automatically, and increased again when the temperature dropped. This process operated continuously, allowing operation under a constant, pre-set temperature. More details of this MWR can be found in our previous studies [24].

2.3. Experimental procedure

For obtaining precise experimental results at a constant temperature, the duration of the sample heated from the room temperature to the target temperature should be as short as possible to decrease the occurrence probabilities of other reactions such as dehydration and torrefaction. Because absorption of microwave of pure straw is weak, using pure straw as sample directly will not achieve the above requirement. So the microwave receptor is necessary, which is what is used in order to heat up the raw biomass. According to previous studies [25,26] and our previous experiment tests, the pyrolysis residue is used as the microwave receptor. The temperature used for pyrolysis is 400 °C, 500 °C, and 600 °C, chosen according to the former researches on biomass pyrolysis [27].

For each trial, 10 g (precision to 1 mg) of shredded and sieved wheat straw mixed with 5 g pyrolysis residue was placed in a quartz crucible. The additives and wheat straw powder were weighed separately, then they were put into a container and mixed by a stirrer until the additives and wheat straw powder was mixed uniformly. After the quartz crucible was correctly positioned and the quartz tube was tightly sealed, a constant flow (3 L/min) of carrier gas (N_2) was purged into the reactor. When the purging was sufficient to produce an anoxic state, the microwave generator was turned on and switched to the designated power.

As the reaction proceeded, the reaction temperature measured by thermocouple was recorded at periodic intervals. When the temperature achieved the target value, the temperature control function commenced, and the sample pyrolyzed at a constant temperature. After the designated reaction time passed, the microwave generator was turned off, but the carrier gas kept purging until the solid residue (char) was self-cooled to below 100 °C. The gas collector was then carefully removed and sealed. The solid residue was placed in a desiccator and weighed after it reached room temperature. The yield of solid products was calculated excluding the mass of additives, and the mass of additives was considered to be unchanged during the experiments. To protect the experimental apparatus, the sample was only heated for 10 min after the temperature of the sample reached the set temperature. If experiments under a higher temperature would be carried out, then the temperature control function could simply be canceled. For guarantying the accuracy of experiments, every condition was repeated two times and the result was the average value of these two experiments.

2.4. Analysis of products

To determine the main composition of the gas products, online FTIR analysis of the gas products was carried out. The condensed and dried gases were directly introduced into the FTIR analyzer (DX4000, GASMET, Finland). The carrier gas flow was 3 L/min, designed to make gas products as fast as possible at a lower

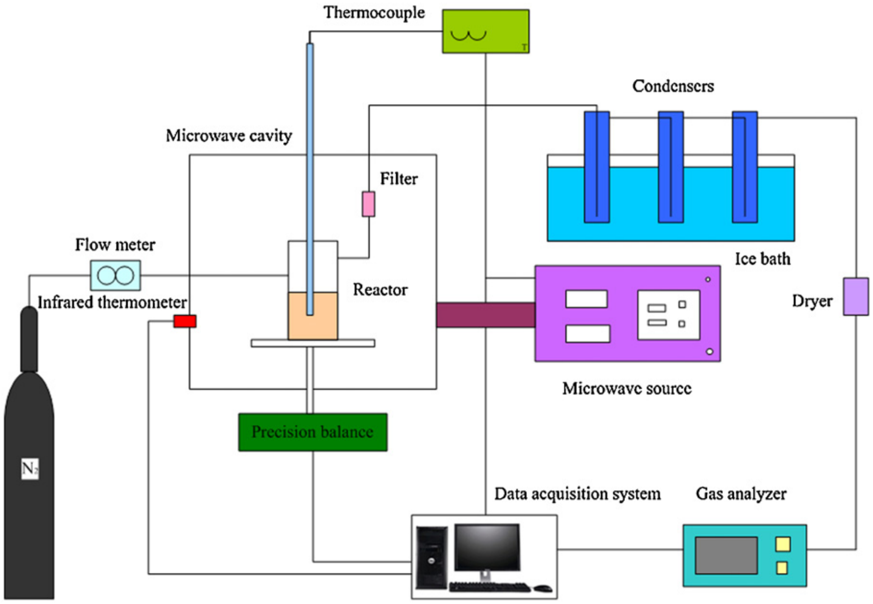

Fig. 1. Schematic diagram of experimental apparatus.

temperature and reduce the occurrence probability of the secondary decomposition of the primary products. The temperature in the test chamber and the gas transmission pipeline was kept at 180 °C to prevent the condensation and absorption of the semi-volatile gas products. The volume flow of gas products was determined with a flow meter at the outlet of the FTIR analyzer. Then the weight of gaseous products was obtained by the total volume multiplied by the average molecular weight and the weight of liquid products was obtained by subtraction of gas products and solid products from the total weight of sample.

For validating the results obtained by FTIR and determining the concentration of H_2 in the gases products, the gases flowing out of FTIR analyzer was analyzed by an Agilent 6890N gas chromatography/dual thermal conductivity detector with a GDX401 capillary column and 5A molecular sieve. The temperature of the injector, oven, and detector was 100, 80, and 150 °C, respectively. The flow rate of the carrier gas was 15 mL/min without splitting.

The solid products were ground to fine particles using a mortar and subjected to FTIR spectrometer which was used in Section 2.1. The IR spectra were between 4000 and 600 cm^{-1} with a resolution of 4 cm^{-1}.

3. Results and discussion

Both the heating rate and the final temperature are the key parameters in biomass pyrolysis [28]. For microwave pyrolysis, the heating rate of sample depends on the microwave power used. In order to investigate the influences of heating rate on the microwave pyrolysis, different power was used to heat the sample and steady its temperature at 500 °C. For investigating the impact of final temperature on the microwave pyrolysis, the pyrolysis experiments of sample which have heated and pyrolyzed at constant temperatures (400, 500, 600 °C) were designed.

3.1. Microwave pyrolysis at a constant temperature

3.1.1. Microwave pyrolysis at a constant temperature using no additives

The product distribution of microwave pyrolysis of wheat straw at final temperature 500 °C using no additive with different microwave powers is shown in Fig. 2. It can be seen that the yield of solids decreased from approximately 50 wt.% to 40 wt.%, and the yield of gas products increased from 20 wt.% to 30 wt.% when the microwave power increased from 400 W to 600 W. However, the yield of liquid products increased slightly. The composition analysis of gaseous products listed in Table 2 shows that the increase of H_2 was approximately 20 wt.%, while the yields of CO_2 and CO dropped significantly when the power changed from 400 W to 600 W.

The reason is that the final temperatures are all at 500 °C, but the heating rates of sample are different, which depends on the microwave power. The higher the microwave power, the higher the heating rate. In the present experiments, the different heating rates of sample at different microwave powers resulted in the different heating time, and the duration of sample in different temperature areas changed correspondingly. As we all know slow pyrolysis of biomass is apt to produce more char, and CO_2 and CO are easily produced at low temperature. So the high production of solid products and the high contents of CO_2 and CO at 400 W may be

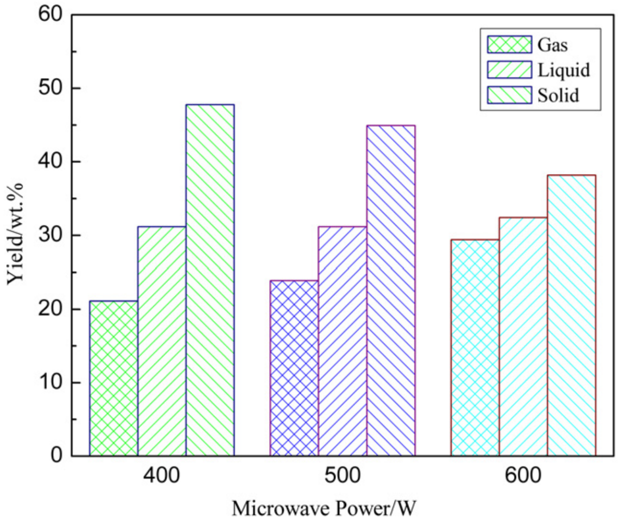

Fig. 2. Products distribution at 500 °C and different microwave powers.

Table 2
Compositions of gas products at 500 °C using different microwave powers.

Power (W)	CO_2 (vol.%)	CO (vol.%)	CH_4 (vol.%)	C_{2+} (vol.%)	H_2 (vol.%)
400	32.8	34.4	7.9	3.3	21.6
500	32.2	32.3	7.7	2.9	24.9
600	22.9	27.3	5.7	2.0	42.1

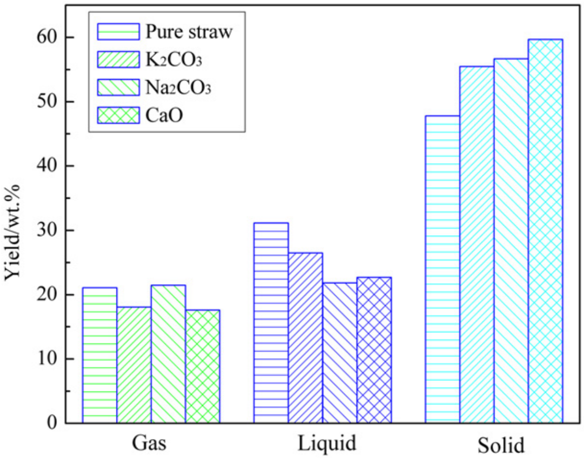

Fig. 3. Products distribution of microwave pyrolysis at 400 W, 500 °C and adding ratio 1:10 of additives.

attributed to the low heating rate, and the shorter the duration at low temperature, the less the production of CO_2 and CO [29].

3.1.2. Microwave pyrolysis at a constant temperature using additives

The product distribution of microwave pyrolysis at 400 W, 500 °C, and with 1 g of additives is shown in Fig. 3. It can be seen that the yield of solids increased by approximately 10 wt.% after adding additives, and the highest amplitude increase was obtained by adding CaO. This result was opposite to that of conventional pyrolysis. The reason is the non-absorption of microwave of additives. After adding the additives, the heat and mass transfer resistance between the biomass particles increased, which limited further pyrolysis of more biomass. In addition, the catalytic ability of these additives was not fully displayed because these catalysts worked better at higher temperature [30]. As a result, the yield of gas products was almost unchanged, whereas the yield of liquids decreased.

The product distribution of microwave pyrolysis at 400 W, 500 °C, and with addition of different ratios of K_2CO_3 is shown in Fig. 4. We can see that the yields of gases and solids showed different behaviors at different addition ratios of K_2CO_3. The yield of gas products at the addition ratio of 1:10 was lower than that of pure wheat straw. However, the gas products at the addition ratio of 1:5 were higher than that obtained with pure wheat straw.

At the prime tense, the increase of gases was attributed to the catalysis of K_2CO_3, which is often used to be catalyst for tar transformation and more CO, H_2 and CH_4 are obtained. However, the composition analysis in Fig. 5 revealed that the increased part of gas products at the addition ratio of 1:5 was almost entirely CO_2 and CO, indicating that the increase of gas products was not produced by K_2CO_3. The reason is the catalysis of K_2CO_3 is weak at the low temperature, and addition of K_2CO_3 increase the heat and mass transfer resistance between particles. As a result, at low temperatures, addition of K_2CO_3 would postpone the pyrolysis reactions [30].

The compositions of gas products at 400 W, 500 °C, and with different additives are shown in Fig. 5. It can be found that the content of CO_2 in gas products decreased more than 10 vol.%, and addition of CaO decreased the content of CO_2 most, approximately 35%. The reason is absorption of part of CO_2 by CaO and formation of $CaCO_3$. As the decomposition temperature of $CaCO_3$ exceeded 700 °C, but the final pyrolysis temperature in our experiments was only 500 °C; hence, the absorbed CO_2 was not re-released. Consequently, the minimum of the gas products, the maximum of the remaining solids, and the minimum concentration of CO_2 were obtained after adding CaO. As a result, CaO could then be used as additives to absorb CO_2 to produce pure hydrogen in microwave pyrolysis or gasification.

Based on the IR spectrogram of solid coke shown in Fig. 6, the main functional groups of coke are OH (3400–3200 cm^{-1}, alcohol), C—H (2936–2916 cm^{-1}, olefin), O═C═O (2350–2310 cm^{-1}), C—C (1475–1435 cm^{-1}, alkane, R-CH_3, R′-CH_2-R′ or aromatic ring), CO—(C) (1275 cm^{-1}), CO—(H) (1050 cm^{-1}) and C—H (865–810 cm^{-1}, C_{5+} olefin or aromatic ring).

From Fig. 6, it can be seen that the OH was removed, and its IR absorption band decreased rapidly. Moreover, the IR absorption

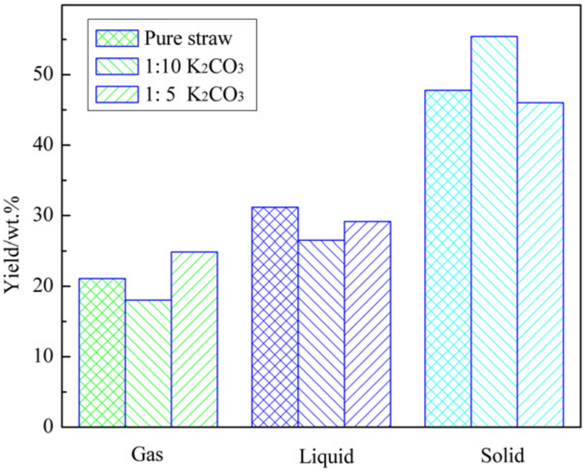

Fig. 4. Products distribution at 400 W, 500 °C and adding different additives.

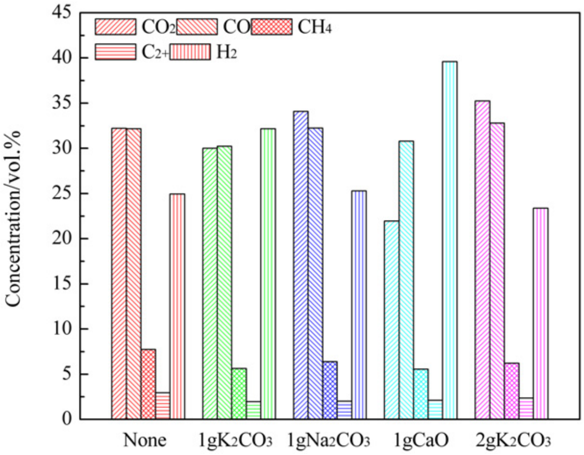

Fig. 5. Products composition at 400 W, 500 °C and adding different additives.

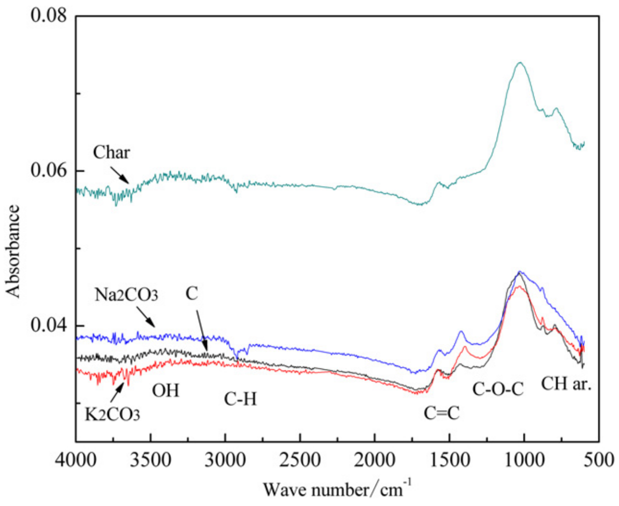

Fig. 6. IR spectrogram of solids obtained using different additives.

Table 3
Pyrolysis product distribution of constant temperature pyrolysis (400, 500, 600 °C) and pyrolysis after canceling of temperature control (400–, 500–, 600–).

Temperature (°C)	Gas (wt.%)	Liquid (wt.%)	Solid (wt.%)
400–	34.7	34.1	31.2
400	17.7	25.2	57.1
500–	36.2	32.6	31.2
500	21.1	31.1	47.8
600–	35.5	34.4	30.1
600	22.3	30.7	47.0

band of the C—H bond (olefins) decreased quickly, which is attributed to the rupture of weak alkyl C—H bonds. This temperature increase favors the formation of gaseous hydrocarbons [31]. As a result, the yields of CH_4, C_2H_6, C_2H_4, and so on increased, whereas the content of element H in the coke decreased markedly. Also, the IR absorption band of O=C=O (CO_2) in coke was removed rapidly, indicating the rupture of O=C=O and formation of CO_2. The C=O (1700 cm^{-1}) bonds within the samples were easily broken to form CO and CO_2 because no IR absorption band of the C=O bonds existed within all the solid products.

3.2. *Microwave pyrolysis after canceling of temperature control*

For investigating the pyrolytic characteristics of wheat straw better, the pyrolysis of wheat straw at constant temperatures (400, 500, 600 °C) are carried out during 10 min firstly, and followed by a stage of constant power with increasing temperature, since more and more char, which absorbs microwave radiation, is being formed. The operating conditions are as follows: the sample was heated to 400, 500, 600 °C using 400 W and maintained for 10 min. Then, the temperature control was canceled, and the sample was heated for 10 min at constant power 400 W.

Table 5
Pyrolysis product distribution of pyrolysis after canceling of temperature control (500–).

Sample	Gas (wt.%)	Liquid (wt.%)	Solid (wt.%)
None	36.2	32.6	31.2
1 g K_2CO_3	37.9	32.9	29.2
2 g K_2CO_3	39.1	34.0	26.9

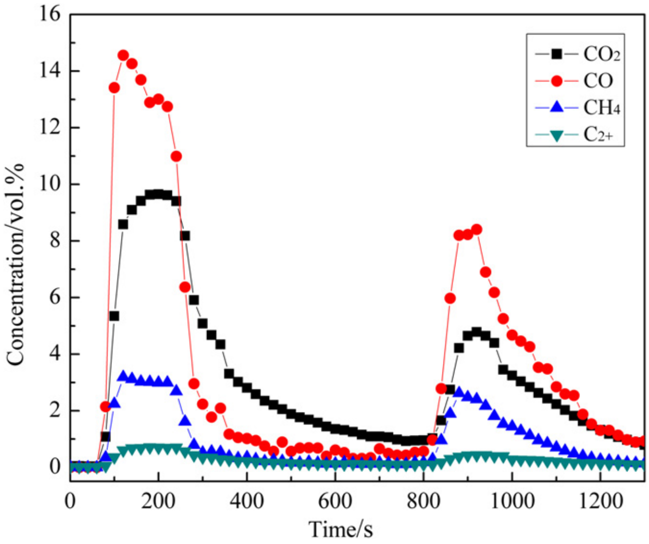

Fig. 7. Gas release profiles of sample heated at 400 W, 500 °C, and with a heating stage of constant power 400 W.

3.2.1. *No additives*

The comparisons of products distribution between constant temperature pyrolysis and continued heating after constant temperature pyrolysis are listed in Table 3. We can see that the yield of solid products continued to decrease significantly by up to 25 wt.%. The yield of liquid products increased slightly, and the yield of gas products had a substantial increase, indicating continued decompose of sample when heated to the high temperature, favorable for conversion of residues to char and gases. The final temperature of sample after canceling temperature control can reach about 800 °C because more char was produced, which absorbs microwave strongly.

The comparison of gas composition between constant temperature pyrolysis and continued heating after constant temperature pyrolysis are listed in Table 4. The CO content increased, and the CO_2 content decreased. In particular, the concentration of H_2 clearly increased because the high temperature promotes the secondary cracking of hydrocarbon molecules and the CH_4 cracking reaction [21]. The yields of syngas (CO + H_2) exceeded 75 vol.% in the total gas products, indicating that microwave pyrolysis had an advantage in producing hydrogen and syngas.

Fig. 7 shows the gas release profiles of sample heated at 400 W, 500 °C for 10 min, and with a heating stage of constant power 400 W

Table 4
Products composition of constant temperature pyrolysis (400, 500, 600 °C) and pyrolysis after canceling of temperature control (400–, 500–, 600–).

Temperature (°C)	CO_2 (vol.%)	CO (vol.%)	CH_4 (vol.%)	C_{2+} (vol.%)	H_2 (vol.%)
400–	18.7	24.7	5.8	1.8	49.0
400	32.8	34.4	7.9	3.3	21.6
500–	18.5	21.3	5.3	1.5	53.4
500	32.3	32.2	7.7	2.9	24.9
600–	15.9	18.3	4.6	1.5	59.7
600	23.0	27.2	5.7	2.0	42.1

Table 6
Compositions of gas products of pyrolysis after canceling of temperature control (500–).

Sample	CO_2 (vol.%)	CO (vol.%)	CH_4 (vol.%)	C_{2+} (vol.%)	H_2 (vol.%)
None	18.5	21.3	5.3	1.5	53.4
1 g K_2CO_3	18.6	21.3	4.2	1.6	54.3
2 g K_2CO_3	16.7	25.9	3.8	1.4	52.2

for 10 min. Two distinct gas release peaks appeared at the concentration curve of gases. Also, it could be inferred that the sample began to pyrolyze after microwave heating for 2 min. Before reaching the set temperature, most volatiles had been released, and the weight loss was over 50 wt.% of the total weight of sample. Consequently, the first peak corresponded to the release of most volatiles. The concentration of gases rapidly declined when the temperature of sample was kept at 500 °C, but the volatiles were not released completely due to the low temperature. When the temperature of the sample was allowed to rise from 500 °C to approximately 800 °C, the residual volatiles were fully released, and the second gas release peak appeared.

3.2.2. Adding K_2CO_3

The product distribution and composition of gas products obtained by continued heating after constant temperature (500 °C) pyrolysis are listed in Tables 5 and 6. It can be seen that the conversion rate of solid products was improved with the increase of addition ratio of K_2CO_3, which was attributed to the stronger catalysis of K_2CO_3 at high temperatures. A comprehensive consideration of the results shown in Fig. 3 revealed a decrease in the solids yields from about 55 wt.% to 29.2 wt.%. This result fully emphasizes the important effect of temperature on the pyrolytic characteristics of microwave pyrolysis.

From Table 6, we can see that the CO content increased from 21.3 vol.% to 25.9 vol.%, the H_2 content had almost no change and the CO_2 and CH_4 content decreased slightly, which was consistent with the previous studies [32,33]. Such decrease could be due to the promotion of the gasification reaction between carbon and CO_2, and the dry reforming and cracking reaction of CH_4, which led to more CO and H_2 produced respectively, at high temperature when K_2CO_3 was used as additives. The reactions mentioned above caused the increase in the total gas production. In addition, as more gases and hydrogen was produced at high temperature, the H_2 content in gas products was above 50 wt.%, and the CO content in gases was lower than that obtained at 500 °C shown in Fig. 4.

4. Conclusions

Temperature and additives are found to affect the pyrolytic characteristics of microwave pyrolysis significantly. The increase in microwave power, leading to a higher heating rate, elevates the gas yield. High temperature is favorable to the production of gaseous products and hydrogen. The presence of additives significantly increases the yield of gaseous products at the expense of the liquid and solid products. The catalysis of K_2CO_3 is weak at the low temperature, but at high temperature, addition of K_2CO_3 promotes the gasification reaction between carbon and CO_2, and the dry reforming and cracking reaction of CH_4. Due to absorption of CO_2, CaO may be used as absorbent of CO_2 for the production of pure hydrogen or syngas through microwave pyrolysis and/or gasification.

Acknowledgements

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